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Method of Forging High-Purity Titanium for Sputtering Target

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ABSTRACT

Objective: To obtain uniform thickness of sputtering film by providing a forging method for high-purity titanium material used for manufacturing high-quality Ti targets.

Content: High-purity Ti for making Ti sputtering targets is forged in combination of swaging and upsetting at a temperature above the phase transformation point, so that the forging ratio is at least 5. Then forging with combination of swaging and upsetting is performed below the phase transformation point to obtain a forging ratio of at least 5.

Claims

A forging process that is applied on high-purity titanium used in manufacturing Ti sputter targets as follows. Combination of swaging with upsetting is performed at a temperature above the phase transformation point as the first forging stage. Combination of swaging with upsetting is performed at a temperature below the phase transformation point as the second forging stage? The forging ratios for both stages are larger than 5. The two-stage forging process is performed at least once on high-purity titanium.

Details of Invention

Industrial Application

This invention provides a forging process for high-purity Ti material used in manufacturing titanium sputter targets.

Conventional Technology

In semiconductor industry, high- purity titanium films are formed by sputtering titanium targets. The high-purity titanium sputtering targets are usually manufactured by a procedure consisting of casting, forging, rolling and heat treatment.

For the uniformity of the sputtering film thickness, it is necessary to refine the grain size of the target. Therefore, rolling and heat treatment is adopted to control the grains size, while forging and rolling are performed for shape control of the targets.

In addition, conventional forging method is based on the forging the whole ingot. For manufacturing titanium sputtering targets, rolling is performed to obtain appropriate shape of the targets based on the particular geometry of ingot. The cast structure is partially broken down during the process. For example, in the Japanese patent 62-280639, which is not specially for the manufacturing target, the forging of titanium was executed by combination of swaging with upsetting repeatedly. In this process, the forging ratio at a temperature above and below the phase transformation point are 5-8 and 3-4, respectively. As a result of this process, grain size is refined.

Problem Solved by This Invention

The process of combination of swaging with upsetting can effectively break down the cast structure of high-purity titanium. The subsequent rolling and heat treatment on high purity Ti in manufacturing Ti targets has little effect on the uniformity of sputtered film thickness. Therefore the practical function of this process is limited. The objective of this invention is to provide a forging method for high-purity Ti material used for manufacturing Ti targets that provides very uniform Ti thin film.

Approach for Solving the Problem

In order to manufacture Ti target that provides high uniformity of film thickness, the inventors performed series of experiments with emphasis on forging process. The results show that the combination of swaging with upsetting, particularly at a temperature below the phase transformation point is very important in manufacturing titanium target. The

reason that repeated forging has no significant effect on sputtering performance as reported by the Japanese patent 62-280639 is insufficient amount of forging at the temperature below the phase transformation point. Forging at a temperature below the phase transformation point is as important as forging at a temperature above the phase transformation point. Therefore, titanium sputtering targets manufactured by the forging process suggested by patent 62-280639 was not very effective on improving the uniformity of film thickness.

Based on the experimental results, this invention adopts a two-stage forging process with the following characteristics for high-purity titanium material used in manufacturing Ti sputtering targets. Combination of swaging and upsetting is applied at a temperature above the phase transformation temperature in the first stage of forging to achieve a forging ratio of more than 5. And then, combination of swaging and upsetting is applied again at a temperature below the phase transformation temperature in the second stage of forging to achieve a forging ratio more than 5.

Description of Invention

4N5 grade(99.995%) high-purity titanium was used in this invention. The forging ratio is the summary of ratios of cross-section areas after each swaging and upsetting forging as illustrated in Figure 1. The deformation ratios of this invention are more than 5 for both the first and the second forging stages.

Since the purpose of the first forging stage is to break down the cast structure, it is performed at a temperature above the phase transformation point at which titanium has good formability. However, if the forging temperature is higher than necessary, more oxidation of Ti will be promoted. Therefore, the temperature for the first forging stage is preferably 900-950°C. If the forging ratio is less than 5, the cast structure will break down insufficiently. On the other hand, large forging ratio in the first forging stage does not provide the benefit that the second forging stage provides, and larger forging deformation means higher cost in production. Therefore, it is desirable to keep the forging ratio less than 10 above the phase transformation point and it is sufficient to perform such forging once.

The purpose of the second forging is to accumulate the amount of deformation. The first forging was performed at a temperature above the phase transformation point at which titanium has good formability. Though it can break down the cast structure, the amount of deformation can not be accumulated in the first forging. The accumulation of the deformation during the second forging promotes the recrystallization after subsequent rolling and heat treatment. The grain refining of the titanium target is benefit to the uniformity of film thickness. In order to accumulate the amount of deformation, second forging is performed at a temperature bellow the phase transformation temperature at which the formability of titanium is not good. Cracks may occur if the forging temperature is too low. Therefore, the lower temperature limit should be higher than 500°C. It is necessary to have the forging ratio higher than 5, preferably higher than 10, and ideally 20.

From the economic point of view, forging ratio of 10-20 is desired, and 15-20 is preferred. It is desirable that two forging cycles, preferably three forging cycles are performed.

Swaging combined with upsetting forging are adopted in both the first and second forging stages. The cast structure is broken down and the uniformity of microstructure is promoted due to the increase of the forging ratio in first forging stage. The grain size is refined during the subsequent rolling and heat treatment due to the increase of the forging ratio in the second forging stage.

Examples

The effect of this invention can be seen from the following examples. A high-purity titanium (4N5) ingot in which the impurity contents are shown in Table 1 was forged according to the conditions shown in Table 2. The microstructure of the forging billets was investigated at cross-sections perpendicular to the directions of reduction. The qualification standard is that the largest grain size should be less than 5 mm.

Table 1 Unit: ppm

Fe	Cr	Ni	Na	K	Th	U	O
10	3	2	<0.1	<0.1	<0.001	<0.001	250

Table 2

No.	Heating to 1050-950°C Forging Ratio	Heating to 1050-950°C No. of Cycles	Heating to 860-600°C Forging Ratio	Heating to 860-600°C No. of Cycles	Microstructure Qualification	Grain Size (μm)	Distribution of Film Thickness (%)
1	9	--	--	--	Negative	*	12
2	5	--	4	--	Negative	*	11
3	14	--	5	1	Negative	50	10
4	15	1	--	--	Positive	*	12
5	17	2	--	--	Positive	*	12
6	7	1	8	--	Positive	*	11
7	7	1	10	1	Positive	30	8
8	7	1	14	2	Positive	20	7
9	7	1	20	3	Positive	10	5
10	5	1	5	1	Positive	50	9
11	10	1	15	1	Positive	15	7
12	17	2	10	1	Positive	30	8

* Contains unrecrystallized grains.

All the billets were rolled with deformation of 50% at 300°C, and then heat treated at 500°C to produce blanks of sputtering targets. The grain sizes were measured at the cross-section. The distributions of film thickness were measured after the sputtering deposition with these targets. The average thickness of the sputtering films was 50 nm. The distributions of film thickness, (maximum film thickness−minimum film thickness) / (average film thickness× 2) ×100%, are shown in Table 2.

In case of example No. 1, forging was performed without the combination of swaging with upsetting for either the first or the second forging stage. The structure of forging is not satisfactory. After rolling and heat treatment, unrecrystallized grains remained in the titanium target. The corresponding distribution of the film thickness is 12%. In the case of examples No.2 and No.3, the targets experienced two forging stages and the sputtering film thickness was not uniform as in the case No.1.

In case of examples No.4 and No.5, although combination of swaging with upsetting were performed in the first forging stage and a satisfactory microstructure was obtained after the forging, the distribution of sputtering film thickness was not improved due to the unrecrystallized grains retained in the targets. In case of example No.6, although the target experienced the second forging and the forging ratio was large enough in the first forging, the uniformity of sputtering film thickness is still not good due to insufficient deformation in the second forging stage.

In case of examples No.7-12, not only the combination of swaging with upsetting was performed in both the first and the second forging stages, but also the amount of deformation was sufficient. Therefore, the billets had satisfactory microstructure, and the targets had fine grains without unrecrystallized grains. As a result, the uniformity of the film thickness increased significantly.

Effect of the invention

It is evident from the results shown above that the forging process of this invention, that is a first forging stage at a temperature above the phase transformation point followed by a second forging stage with a significant amount of deformation at a temperature below the phase transformation point, can provide high-quality targets for good uniformity of film thickness distribution. The improvement in film uniformity will promote high level of integration in semiconductor industry.

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【0019】No. 4, 5では、1次鍛造加工で鍛伸と据え込みの組み合わせを行ったので、鍛造材のマクロ組織は合格となったが、ターゲットには未再結晶粒が残り、スパッタ膜厚分布はNo. 1の場合と変わらなかった。No. 6のように2次鍛造加工を行なっても、それが不十分であると、1次鍛造加工での加工度を大きくしてもスパッタ膜厚はそれほど均一化されない。

【0020】これらに対し、No. 7～12では1次鍛造加工および2次鍛造加工で鍛伸と据え込みの組み合わせを行い、かつその2次鍛造加工で十分な加工を行ったので、鍛造材のマクロ組織は合格となり、ターゲットでも

未再結晶粒のない微細な結晶粒が得られ、その結果、スパッタ膜厚の分布は大幅に均一化された。

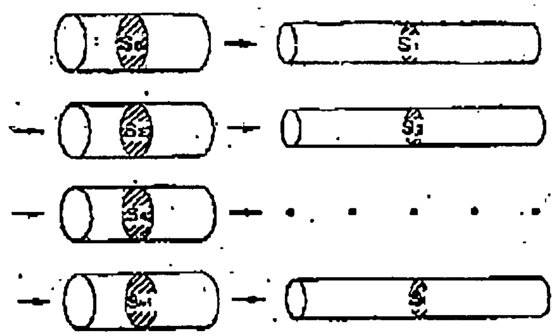
【0021】

【発明の効果】以上に説明した通り、本発明の高純度チタン材の鍛造方法は、変態点以上の1次鍛造加工に続けて変態点以下で十分な2次鍛造加工を行うことにより、スパッタ膜厚の均一性に優れた高品質なスパッタリング用チタンターゲットを提供でき、半導体ディバイスの高集積化等に寄与する。

【図面の簡単な説明】

【図1】鍛伸と据え込みの組み合わせ加工およびその加工での鍛錬成形比を示す図である。

【図1】



Forging Ratio (鍛錬成形比) $= \frac{S_0}{S_1} + \frac{S_2}{S_1} + \frac{S_2}{S_3} + \frac{S_4}{S_3} + \cdots\cdots + \frac{S_{i-1}}{S_i} + \frac{S_{i+1}}{S_i}$

＊ 但し、鍛造回数が奇数の時は $\frac{S_{i+1}}{S_i}$ を省略し、i には鍛造回数を代入

偶数 ＝ (鍛造回数) －1 を i に代入

Figure 1